PRODUCTION OF SILICON SINGLE CRYSTAL WAFER

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Abstract

PROBLEM TO BE SOLVED: To produce silicon single crystal wafers that have excellent device properties via the Czochralski process.
SOLUTION: (1) Relating to the production process for silicon single crystal with an oxygen concentration of $<= 13 \times 10^{17}$ atoms/cm^3 by the Czochralski process, the latent area of the oxidation-induced lamination defects revealing in a ring form inside the crystal face is controlled to 70% to 0% of the crystal diameter, and the V/G value is controlled to be $>=$ a prescribed limit value in the radial direction except the outermost periphery, when the pulling-up rate is set to V (mm/min) and the temperature ingredient in the direction of the lifting up shaft is set to G (deg.C/mm). (2) The pulling up speed is more increased to $>=1.0$ mm/min, when the silicon single crystal of $>=13 \times 10^{17}$ atoms/cm^3 oxygen concentration is produced. In the production of slicon single crystal wafers, the temperature ingredient G in the crystal in the lifting up direction is calculated by the heat transfer calculation and the V/G limit value is made to 0.20 mm^2 / deg.C. min and the operation is controlled so that the V/G value may exceed this value.

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(57)[SUMMARY]

[SUBJECT] The silicon-single-crystal wafer which was excellent in the device characteristic by the Czochralski method is manufactured.

[SOLUTION] (1) It is the method of manufacturing the silicon single crystal whose oxygen density is under $13*IO17 atoms/cm3$, by the Czochralski method, comprised such that it is made to become the range whose radius of the potential area of the oxygen induced stacking fault which appears in the shape of a ring in the crystal plane is 70% - 0% of the crystal radius.
And, raising speed is set to V (mm/min).
  When setting the gradient degree of crystal inside temperature in the raising axial direction to G (degree C/mm), V/G value is controlled above a

predetermined threshold value in the direction position of the path except for the most external circumference of the crystal.
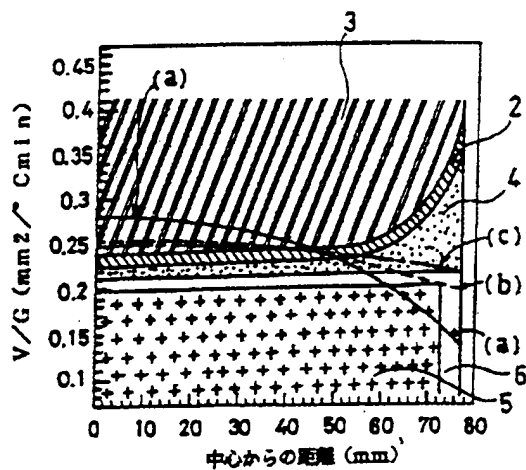
The manufacturing method of the silicon-single-crystal wafer characterized by the above-mentioned.

(2) When the silicon single crystal whose oxygen density is more than 13*IO17atoms/cm3 is manufactured, furthermore raising speed is made more than 1.0 mm / min.

The manufacturing method of the silicon-single-crystal wafer characterized by the above-mentioned.

In the manufacturing method of the above-mentioned silicon-single-crystal wafer, gradient degree G of crystal inside temperature in the raising axial direction is computed by the thermal rating.

With the threshold value of V/G being 0.20 mm2/degree C*min, it is desirable to set the V/G value higher than that.

[translation of Japanese text in Selection Diagram]
horizontal axis: distance from center (mm)

[CLAIMS]

[CLAIM 1] A manufacturing method of the silicon-single-crystal wafer, in which the method of manufacturing the silicon single crystal whose oxygen density is under 13*IO17atoms/cm3, by the Czochralski method, comprised such that it is made to become the range whose radius of the potential area of the oxygen induced stacking fault which appears in the shape of a ring in the crystal plane

is 70% - 0% of the crystal radius.

And, raising speed is set to V (mm/min).

When setting to G (degree C/mm) the gradient degree of crystal inside temperature in the raising axial direction in the specific temperature region within the limits up to 1300 degrees C from the silicon melting point, v/G value (mm2/degree C*min) is controlled above a predetermined threshold value in the direction position of the path except the most external circumference of the crystal.

[CLAIM 2] A manufacturing method of the silicon-single-crystal wafer, in which the method of manufacturing the silicon single crystal whose oxygen density is more than 13*IO17atoms/cm3, by the Czochralski method, comprised such that it is made to become the range whose radius of the potential area of the oxygen induced stacking fault which appears in the shape of a ring in the crystal plane is 70% - 0% of the crystal radius.

And, raising speed is set to V (mm/min).

When setting to G (degree C/mm) the gradient degree of crystal inside temperature in the raising axial direction in the specific temperature region within the limits up to 1300 degrees C from the silicon melting point, v/G value (mm2/degree C*min) is controlled above a predetermined threshold value in the direction position of the path except the most external circumference of the crystal.

Furthermore raising speed is used at more than 1.0 mm / min.

[CLAIM 3] A manufacturing method of the silicon-single-crystal wafer of Claims 1 or 2, in which the above-mentioned gradient degree G of crystal inside temperature is computed from the mean value of the temperature gradient (1300 degrees C and 1400 degrees C) by the thermal rating, and with the above-mentioned threshold value being 0.20 mm2/degree C*min, v/G value is made more than that.

[DETAILED DESCRIPTION OF INVENTION]

[0001]

[INDUSTRIAL APPLICATION] This invention becomes as follows about the

manufacturing method of the silicon single crystal used as a semiconductor device.

It is related with the method of specifically manufacturing the quality silicon-single-crystal wafer without detrimental Grown-in defect by the Czochralski method (henceforth, the CZ process).

[0002]

[PRIOR ART] As the method of carrying out the growth of the silicon single crystal used for a semiconductor material, there are various methods.

However, the CZ process is used abundantly among them.

When the silicon-single-crystal wafer manufactured by the CZ process receives a heat oxidation treatment (for example, 1000-1200 degrees-C * 1-10 hours) in an acid atmosphere, the oxygen induced stacking fault (henceforth OSF ring) which appears in the shape of a ring may occur.

Thus the area which OSF ring generates potentially is influenced by the raising speed during growth (mm/min).

If raising speed is made small, the area where OSF ring appears contracts inside from the periphery of the crystal.

If it puts in another way, when carrying out the growth of the single crystal at high speed, the inner-side area of the OSF ring will spread to the whole wafer.

If a growth is carried out at a low speed, the outer-side area of the OSF ring will spread to the whole wafer.

[0003] In the inner-side area of the OSF ring of the single-crystal wafer manufactured by the CZ process, the octahedron void supposed to be the aggregate of pores forms.

When manufacturing MOS type LSI, in the gate active region, the breakdown-voltage characteristic of a gate oxide film is degraded, and a poor separation is produced in the element separation area.

Furthermore when using a trench capacitor, causing poor characteristics, such as the punch through between capacitors, is clarified.

(For example, Yamamoto, Hidekazu * Koyama, Hiroshi : Applied physics, 66,662 (1997)).

[0004] Since it corresponds to such a problem, many process is conventionally

proposed.

First, in the Provisional-Publication-No. 2-267195 gazette, when the silicon single crystal of diameter 100 mm or more is manufactured by the CZ process, the process of making raising speed below 0.8 mm / min is proposed.

That is, the position at which OSF ring will generate is shrunk from the crystal periphery by setting raising speed at a low speed called below 0.8 mm / min.

It is made to disappear in the main part of a crystal.

According to this process, in the outer-side area of the OSF ring, since the oxide-film breakdown-voltage is good, suppose that an oxide-film breakdown-voltage characteristic can be raised in connection with a reduction of raising speed.

[0005] However, surely, by the process proposed here, the oxide-film breakdown-voltage characteristic becomes satisfactorily.

However, in the about 0.4 - 0.8 mm/min range, generating of oxygen induced stacking fault (henceforth OSF) has raising speed in the area of the OSF ring in connection with a heat oxidation treatment.

Furthermore if raising speed is made smaller than about 0.4 mm/min, the problem of the characteristic deterioration by the transition cluster generated to the outer-side area of the OSF ring will be generated.

[0006] Next in the Provisional-Publication-No. 9-202690 gazette, by wafer area ratio 50 % or more, or the area of the periphery to 30 mm or more, the silicon-single-crystal wafer which is a defect-free area without breakdown-voltage deterioration or poor breakdown-voltage, in order to carry out the growth of this, as opposed to the maximum raising speed with raising speed intrinsic in the reactor, the process manufactured at 80-60% of raising speed is disclosed.

And, according to this process, there is no formation of the potential nucleus of OSF and there is no generating of OSF also by heat oxidation treatment in the device process as mentioned later from it being an equivalent for low-oxygen (17 ppma(JEIDA),13*I017atoms/cm3.

[0007] According to the above-mentioned indication, it is specified that there is no FPD, LSTD, and OSF in the defect-free area without deterioration or defect of the oxide-film breakdown-voltage.

However, there is no description about the transition cluster.

That is, on the above-mentioned manufacture conditions, it is not considered that the transition cluster occurs on the outer-side area of the OSF ring, and means or the process for preventing this is not disclosed, either.

For this reason, the oxide-film breakdown-voltage characteristic of the outer-side area of the OSF ring is favourable.

However, it is clear that it causes poor characteristics, such as PN-junction leak, by generating of the transition cluster.

[0008] Finally, the raising speed at the time of single crystal growth and the temperature gradient within the crystal are controlled by the Provisional-Publication-No. 8-330316 gazette.

The process of extending only the outer-side area of the OSF ring to the crystal whole surface is proposed, without making the transition cluster form.
However, by the process of the proposal, since it pulls up with a distribution of the in-plane temperature gradient limited extremely and conditions are demanded simultaneously, a large-diameter enlargement will be carried out much more from now on, and new improvement is demanded in the silicon growing of a single crystal for which mass production is demanded.

[0009]

[PROBLEM ADDRESSED] This invention is made in view of the problem about the manufacturing method of the silicon single crystal by the above-mentioned conventional CZ process.

While eliminating generating of OSF in the potential generating area of the OSF ring which comes out in plane wafer, generating of the transition cluster in the outer-side area of the OSF ring can be suppressed, and the manufacture conditions permitted are comparatively large.

It aims at providing the manufacturing method of the silicon-single-crystal wafer without a reduction of productivity.

[0010]

[SOLUTION OF THE INVENTION] This invention is made in order to solve the above-mentioned subject.

The manufacturing method of the following (1) and the silicon-single-crystal

wafer of (2) is made into the main point.

[0011] (1) the method of manufacturing the silicon single crystal whose oxygen density is under $13*10^{17}$atoms/cm3, by the CZ process, comprised such that it is made to become the range whose radius of the potential area of the oxygen induced stacking fault which appears in the shape of a ring in the crystal plane is 70% - 0% of the crystal radius.
And, raising speed is set to V (mm/min).
When setting to G (degree C/mm) the gradient degree of crystal inside temperature in the raising axial direction in the specific temperature region within the limits up to 1300 degrees C from the silicon melting point, v/G value (mm2/degree C*min) is controlled above a predetermined threshold value in the direction position of the path except the most external circumference of the crystal.
The manufacturing method of the silicon-single-crystal wafer characterized by the above-mentioned.

[0012] (2) the method of manufacturing the silicon single crystal whose oxygen density is more than $13*10^{17}$atoms/cm3, by the CZ process, comprised such that it is made to become the range whose radius of the potential area of the oxygen induced stacking fault which appears in the shape of a ring in the crystal plane is 70% - 0% of the crystal radius.
And, raising speed is set to V (mm/min).
When setting to G (degree C/mm) the gradient degree of crystal inside temperature in the raising axial direction in the specific temperature region within the limits up to 1300 degrees C from the silicon melting point, v/G value (mm2/degree C*min) is controlled above a predetermined threshold value in the direction position of the path except the most external circumference of the crystal.
Furthermore raising speed is used at more than 1.0 mm / min.
The manufacturing method of the silicon-single-crystal wafer characterized by the above-mentioned.

[0013] In the manufacturing method of the above (1) and the silicon-single-crystal wafer of (2), gradient degree G of crystal inside temperature in the raising axial direction is computed from the mean value of the temperature

gradient (1300 degrees C and 1400 degrees C) by the thermal rating.

With the threshold value of V/G being 0.20 mm2/degree C*min, it is desirable to set the V/G value higher than that.

Here, with a predetermined threshold value, the intention of the limitation which can be controlled so that the transition cluster does not occur in the crystal plane is intended as mentioned later.

[0014] The silicon-single-crystal wafer by which the growth was carried out by the CZ process may generate OSF to the generating area of potential OSF ring with high-temperature heat treatment in the oxidation-property atmosphere in the device process as above-mentioned.

This grows with high-temperature heat treatment of the above oxygen precipitate nucleus, which exists in the generating area of the OSF ring in crystal, and it is for inducing OSF as secondary defects.

The area which OSF generates in the shape of a ring in description of the following of this invention, if it puts in another way, the potential nucleus of OSF only expresses the area which exists in the shape of a ring as "the generating area of the OSF ring", or "the generating position of the OSF ring".

[0015]

[Embodiment] These inventors improve the single crystal growing conditions by the CZ process, in order to attain the objective of this invention.

It limits to a specific range almost centering on the generating area of the OSF ring within the wafer surface.

It perceives making the large area of the wafer enlarge the precipitate promotion area and the defect-free area which exist adjacent to the outer area of the OSF ring, and various examination was performed.

[0016] First, it is related with the generating position of the OSF ring.

Raising speed is set to V (mm/min).

When setting to G (degree C/mm) the gradient degree of crystal inside temperature in the raising axial direction in the specific temperature region within the limits up to 1300 degrees C from the silicon melting point, it was carried out being influenced by the ratio of V/G clearly.

In the manufacturing apparatus which has the identical structure, it depends

on drawing-of-a-single-crystal speed for the radius of the OSF ring.

However, when the hot zone structure varies, even when it is identical raising speed, the radius of the OSF ring differs.

However, the radius of the OSF ring can be defined almost uniquely by controlling pertinently the above-mentioned V/ G value, the generating area of the OSF ring can be adjusted.

[0017] It can measure for the temperature distribution within a crystal required for calculation of the above-mentioned temperature-gradient G (degree C/mm) from measurement or a thermal rating by the following (circled-1) - (circled-3) processes.

First, there is a method of inserting ?heat-transfer-pair? into a crystal and measuring temperature within the crystal by simulation (circled-1).

Moreover, the surface temperature of the specific site of the crystal which can be pulled up (circled-2) is measured optically (for example, by CCD camera).

From the relationship of the measured surface temperature and the above-mentioned observed value, there is also a method of supposing the temperature inside the crystal.

Furthermore, (circled-3) from the temperature in which various measurement is possible, from the physical-property value, there is the process of adding a synthetic heat-transfer analysis and calculating temperature inside the crystal.

(For example, S.Miyahara et al.: J.Cryst.Growth 99,696 (1990)) T. Fujiwara et al.: J.Cryst.Growth 128,275 (1993).

[0018] In this invention, by one of (circled-1) - (circled-3) above, temperature-gradient G (degree C/mm) may be computed.

But, if influence on the temperature distribution by the convection current in melt solution is not reflected when performing heat-transfer analysis, the shape of the solid-liquid boundary surface different from practice will be obtained.

It differs from what also has an actual temperature distribution within the crystal.

Therefore, in order to obtain the exact temperature distribution in the high-temperature part especially in case of heat-transfer analysis, the shape of a solid-liquid boundary surface is measured from the actual crystal, and the boundary condition in the heat-transfer analysis is corrected after this.

The temperature distribution of the axial direction inside the crystal needs to

be calculated.

[0019] These inventors ask for a temperature gradient (1400 degrees C on the same axis parallel to the raising direction, and 1300 degrees C) by the process of the above (circled-3) in case of calculation of temperature-gradient G (degree C/mm).

G (degree C/mm) was computed from these mean values.

Then, V/G value is calculated by the relationship with raising speed V (mm/min), and influence affecting the defective distribution within the wafer surface was investigated.

The calculation process of temperature-gradient G (degree C/mm) demonstrated here does not specify the calculation process of the temperature gradient within the crystal in embodiment of this invention.

Moreover, it is not limited to these, either.

In order to obtain the crystal which the transition cluster does not generate according to calculation of the above-mentioned temperature-gradient G (degree C/mm), the threshold value of V/G becomes 0.20 mm2/degree C*min, and it turns out that V/G value needs to be set higher than that.

That is, if V/G value is made more than 0.20 mm2 / degree C*min, the transition cluster will not occur in the wafer surface.

[0020] However, if the calculation process of temperature-gradient G (degree C/mm) differs, the threshold value of V/G for not generating the transition cluster also differs.

Therefore, the threshold value of V/G which the transition cluster does not generate experimentally is calculated for every calculation process of temperature-gradient G (degree C/mm).

The crystal which the transition cluster does not generate can be obtained by setting V/G value higher on the basis of this.

It demonstrates based on the heat-transfer analysis which investigated influence of V/G value which exerts such an example of a control on the defective distribution within the wafer surface which these inventors carried out.

[0021] diagram 1 makes a horizontal axis the radial direction position from the wafer center.

It is the diagram showing a defective distribution which makes V/G value the

vertical axis, and appears in the wafer surface.

As shown in diagram 1, wafer 1 center in-plane around OSF ring 2, and the inner-side area 3 and the outer-side area are divided.

Furthermore the outer-side area of the OSF ring is divided into the precipitate promotion area 4, the transition cluster area 5, and the defect-free area 6.

In the transition cluster area 5, there is worry about a reduction of the device characteristic.

However, there is no such problem in the precipitate promotion area 4 and the defect-free area 6.

The precipitate promotion area 4 does not have generating of the transition cluster which causes the void defect and PN-junction leak which degrade the oxide-film breakdown-voltage.

However, it is the area where oxygen precipitate happens by low hot-temperature process of about 800 degrees C or less.

Moreover, the defect-free area 6 is an area where the oxygen precipitate by the above-mentioned void defect, the transition cluster, and low hot-temperature process does not happen, either.

[0022] Clearly shown in diagram 1, if V/G value becomes under 0.20 mm2/degree C*min in the direction of the path throughout the whole region, the transition cluster occurs mostly

However, the outer layer of the periphery is left out.

The range of the periphery in which the transition cluster does not generate in this way usually is about 5 mm from the most external periphery.

In the outer-side area of the OSF ring, the precipitate promotion area 4 and the defect-free area 6 spread as V/G value becomes large.

Therefore, in order not to make the outer-side area of the OSF ring generate the transition cluster, V/G value needs to be set up more than 0.20 mm2 / degree C*min in the part except the most external circumference.

[0023] Since the yield of the device which can be manufactured from one silicon-single-crystal wafer is raised on the one hand, it becomes the range whose radius of the OSF ring which appears in the crystal plane is 70% - 0% of the crystal radius.

OSF ring usually generated in the wafer surface has the width of several millimeters - 10 mm.

Therefore, for the radius of the OSF ring becoming 70% of the crystal radius, it is the case where the periphery of the OSF ring corresponds to 70% of the crystal radius.

When the radius of the OSF ring turns into 0% of the crystal radius, it is the case where OSF ring disappears from the main part.

[0024] In order to become the radius of the OSF ring made 70% - 0% of the range of the crystal radius, what is sufficient is just to control raising speed V of the mean at the time of operation in 70% - 40% of the range to maximum raising speed Vmax intrinsic in the manufacturing apparatus by the CZ process, according to these inventors examination result.

Here, with intrinsic maximum raising speed Vmax, if it is exceeded and pulled up and speed is gathered, the outer diameter of the crystal growing will deform, and the speed at which a round shape is not maintained is indicated.

Therefore, maximum raising speed Vmax is a numerical value intrinsic in the manufacturing apparatus.

When the structure of the hot zone changes, it varies.

[0025] diagram 2 is a diagram showing the distribution situation of the defect at the time of making an almost fixed radius generate the radius of the OSF ring within the range of 70% - 0% of the crystal radius.

(a) (b), and (c) of Diagram 2 are differing manufacturing apparatus used. To intrinsic maximum raising speed Vmax, raising speed V of the mean at the time of operation is controlled, and the generating position of the OSF ring is made almost fixed.

By (a), the precipitate promotion area 4 exists adjacent to the outer-side area of the OSF ring 2.

However, the defect-free area 6 and the transition cluster area 5 spread out to the outer side.

On the other hand, there was no generating of transition cluster area 5 by (b) and (c).

[0026] As mentioned above, even when the generating position of the OSF ring is almost fixed, since it originates in the structure of the hot zone of manufacturing apparatus and a distribution of V/G differs, the defective distribution is different.

Inside the above-mentioned diagram 1, the direction position corresponding to (a) of (b), and (c) of the path from the wafer center in diagram 2, the distribution of V/G at the time of mean-value G of the crystal temperature of 1400 degrees C and a 1300-degree C temperature gradient is shown.

In order not to make the outer-side area of the OSF ring generate the transition cluster as shown in (b) and (c) of diagram 2, in the manufacturing apparatus of the specific hot zone structure, it turns out that V/G value needs to be set greater than 0.20 mm2 / degree C*min at the position of the radial direction position except the most external circumference.

[0027] As explained above, the silicon-single-crystal wafer manufactured by the CZ process becomes the range whose radius of the OSF ring is 70% - 0% of the crystal radius.

If the temperature distribution in a hot zone is adjusted so that V/G value of the radial direction position of a crystal may cross the area which the transition cluster does not generate, the transition cluster does not exist in the outer-side area of the OSF ring, and it becomes the wafer of the outstanding quality which does not make the device characteristic reduce.

[0028] The silicon single growing crystal by the CZ process by the way, will contain the oxygen impurity which carries out an elution, from the quartz crucible which accommodates a melting raw material structurally.

When the oxygen content of a single silicon crystal becomes more than 13*IO17atoms/cm3, OSF may be induced in the generating area of the OSF ring with high-temperature heat treatment of the oxidation-property atmosphere in the device process.

Since such OSF causes various device characteristics deterioration, the device yield is made to reduce.

However, in the case where the oxygen density which the silicon single crystal contains is under 13*IO17atoms/cm3, with the above-mentioned heat treatment, OSF is not induced in the generating area of the OSF ring.

Thus that OSF does not actualize, since the precipitate nucleus formed into a single crystal at the time of a growth has a low oxygen density, it cannot grow, and it is because it does not become sufficient size to induce OSF.

[0029] On the one hand, when the oxygen density is more than

13*l017atoms/cm3, it must be assumed that OSF is induced in the generating area of the OSF ring.

However, when the growth of the silicon single crystal is carried out in this case, generating of OSF can be suppressed by using raising speed 1.0 mm / min or greater.

That is, the temperature range on which the oxygen precipitate nucleus in the cooling process at the time of crystal growth which turns into the generating nucleus of OSF in the ring area is formed is 850 degree C from about 1000 degrees C.

By carrying out raising speed 1.0 mm / min or greater, the temperature range in which an oxygen precipitate nucleus is formed can be passed early, and it is because the growth of the potential nucleus of OSF can be suppressed.

[0030]

[Embodiment]   The effect of this invention is demonstrated based on embodiments 1 and 2.

[0031] (Embodiment 1)

CZ reactor in which 18"6" growing of a single crystal by which the quartz crucible and the carbon crucible were installed is possible is used, and the pull-up of a single crystal was performed.

The relative position of the carbon heater of the cylindrical shape and the crucible which were installed around the crucible at this time, 5 mm in thickness which consists of carbon installed around the growing crystal, distance on the end of the radiation shield object of the semicircle conical shape of 200 mm diameter the opening, and the surface of melt solution, comprehensive heat-transfer analysis examines the conditions, such as the heat-insulating-material structure of the perimeter of the heater.

It was pulled up with raising speed V (mm/min), and the ratio with gradient degree G (degree C/mm) of crystal inside temperature of an axial direction, and V/G value were defined.

However, temperature-gradient G was taken as the mean of the crystal temperature of 1400 degrees C, and a 1300-degree C temperature gradient.

[0032] Diagram 3 is a diagram showing V / G (mm2/degree C*min) value of

two sorts of hot zones A and B used in embodiment 1.

Maximum raising speed is 1.8 mm/min at hot zone A, in hot zone B, it was 1.4 mm/min.

[0033] 65kg of high-purity polycrystal silicon is put into the quartz crucible, and the dope of the boron is carried out.

After carrying out the heat dissolution of the polycrystalline silicon, the crystal-growth bearing of diameter 150mm (6") is <100>. An about 10 (OMEGA) resistance factor single crystal was raised using the hot zones A and B

By the growing of a single crystal in hot zone A, raising speed is set to 1.8 mm/min by 100 mm below the ?ridge?.

Raising speed was made to reduce gradually at a fixed ratio so that it may be 0.4 mm/min by 500 mm below the ?ridge?.

Moreover, it makes below the ?ridge? to 1.4 mm/min by 100 mm at the growth in hot zone B.

It pulled up so that it might be set to 0.4 mm/min by 500 mm below the ?ridge?, and speed was made to reduce gradually.

After then, these crystals raise bottom of the shoulder 500 mm or later to 700 mm at the raising speed of 0.4 mm/min, until till stop is performed by the usual process, and the growth was completed.

[0034] The crystal after growth is cut out at 1.5 mm thickness in parallel with the direction of the crystallographic axis.

The dissolution removal of the process distortion is carried out in the mixed-acid solution which consists of HF and HNO_3.

Furthermore it is immersed into rare HF solution, rinsed in ultra-pure water, and then dried after that.

It was under $11 - 13*10^{17}$ atoms/cm3 when the oxygen density between lattices in these samples was measured by FT-IR.

After 800 degree-C *4hr+1000 degree-C *16hr heat-treated these samples in the atmosphere of dry oxygen first, using X-ray topography, it investigated the generating situation of the crystal defect.

Moreover, another sample wafer is immersed into 1000 ppm $CuNO_3$ aqueous solution.

After drying naturally, inside of nitrogen atmosphere, 900 degree-C *20min heat-treated.

Then, it immerses into the mixed-acid solution which consists of HF and HNO3.

After about 100 micrometre etching removal, the X-ray topography investigated the outer-layer silicide layer the distribution of Cu decoration.

[0035] diagram 4 is a diagram showing the distribution situation of the crystal defect observed by the (solid-square) ray topography in embodiment 1.

(a) shows the defective distribution of the crystal by which the growth was carried out in hot zone A. (b) shows the defective distribution of the crystal by which the growth was carried out in hot zone B.

On the vertical axis of diagram 4, raising speed (1.8 mm/min - 0.4 mm/min) in hot zone A and, and raising speed (1.4 mm/min - 0.4 mm/min) in hot zone B are displayed.

[0036] diagram 5 is a diagram showing the radial distribution within the wafer surface of V/G value by the heat-transfer analysis in the raising speed shown in diagram 4.

(a) shows the case where growth is carried out in hot zone A. (b) shows the case where growth is carried out in hot zone B.

From the result of Fig. 4 and 5, when V/G value is more than 0.20 mm2/degree C*min In the outer-side area of the OSF ring, the precipitate promotion area and the defect-free area spread, and it has confirmed that the transition cluster did not occur.

[0037] Furthermore, 1100 degree-C *16hr was heat-treated another sample in the dry oxygen atmosphere, and the oxidation was performed.

After removing the oxide film in HF aqueous solution, a selective etching is performed using light etching liquid for about 5 minutes.

The generating situation of OSF was investigated.

However, generating of OSF was not recognised.

From this result, when the oxygen density was under 13*l017atoms/cm3, it was confirmed that OSF does not occur even when it is the generating area of the OSF ring.

[0038] (Embodiment 2)

CZ reactor in which 6" growing of a single crystal used in embodiment 1 is possible is used.

Furthermore the pull-up of a single crystal was performed.

The growing of a single crystal is performed in 2 sorts of hot zones A and B.

Likewise, maximum raising speed in hot zone A is 1.8 mm/min, and 1.4 mm/min in hot zone B.

Under the same conditions except melting-pot rotation, as an embodiment 1, after raising a single crystal, the generating situation of the defect within this crystal was investigated by the same point.

[0039] diagram 6 is a diagram showing the distribution situation of the defect observed by the same process as an embodiment 1 in an embodiment 2.

(The crystal to which the growth of a) was carried out in hot zone A, and (b) show the defective distribution of the crystal by which the growth was carried out in hot zone B.

The oxygen density between these lattices in crystal was 13 - $15*I017$ atoms/cm3.

The generating situation of the OSF ring and the transition cluster is the same as an embodiment 1 almost, and the significant difference was not recognised.

[0040] Next by the same process as an embodiment 1, the generating situation of OSF in a crystal plane was investigated.

Even when it was the single crystal by which the growth was carried out by any of the hot zones A and B, when raising speed became under 1.0 mm / min as a result, generating of OSF was accepted in the generating area of the OSF ring.

On the other hand, generating of OSF was not recognised in the area in which the growth of the raising speed was carried out above 1.0 mm / min.

When raising speed carries out a growth from the above result 1.0 mm / min above

Even when it was the case where the oxygen density in a single crystal was more than $13*I017$ atoms/cm3, not inducing OSF in the generating area of the OSF ring was confirmed.

[0041]

[EFFECT OF THE INVENTION] Without it makes the outer side of the generating area of the OSF ring within a single-crystal wafer surface generate

the transition cluster according to the process of this invention, it can consider as a defect-free area.

Since the potential nucleus of the OSF ring moreover is not made to form irrespective of the oxygen density contained in crystal, OSF is not made to induce even when it is the generating area of the OSF ring.

The single-crystal wafer in which the crystal defect did not exist, and excellent in the device characteristic, by this can be manufactured.

[BRIEF EXPLANATION OF DRAWINGS]

[FIGURE 1] Let the radial direction position from a wafer center be a horizontal axis.

It is the diagram showing a defective distribution which makes V/G value the vertical axis, and appears in the wafer surface.

[FIGURE 2] The diagram showing the distribution situation of the defect at the time of making an almost fixed radius generate the radius of the OSF ring within the range of 70% - 0% of a crystal radius.

[FIGURE 3] The diagram showing V / G (mm2/degree C*min) value of two sorts of hot zones A and B used in embodiment 1.

[FIGURE 4] The diagram showing the distribution situation of the crystal defect observed by the (solid-square) ray topography in embodiment 1.

[FIGURE 5] The diagram showing the radial distribution within the wafer surface of V/G value by the heat-transfer analysis in the raising speed shown in diagram 4.

[FIGURE 6] The diagram showing the distribution situation of the crystal defect observed by the (solid-square) ray topography in embodiment 2.

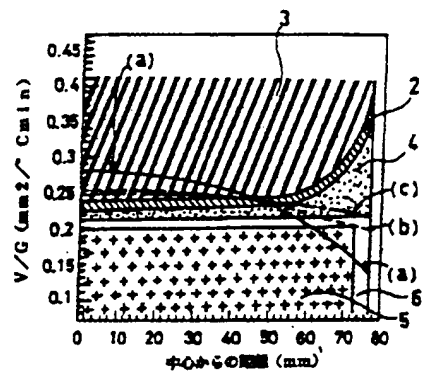## [EXPLANATION OF DRAWING]

1: Wafer, 2: OSF ring
3: The inner-side area of the OSF ring
4: Precipitate promotion area

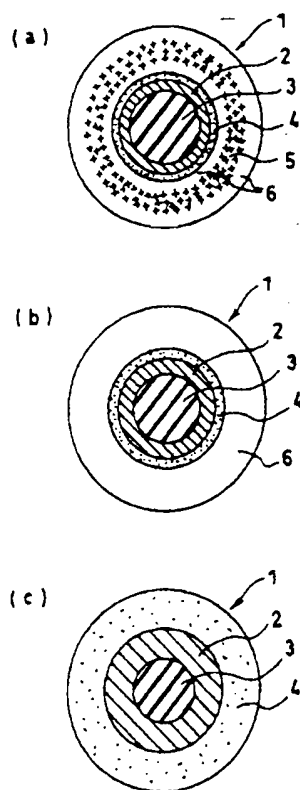5: The transition cluster area

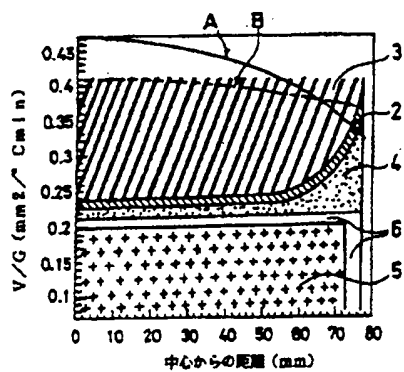6: Defect-free area

[FIGURE 1]

[translation of Japanese text in Figure 1]

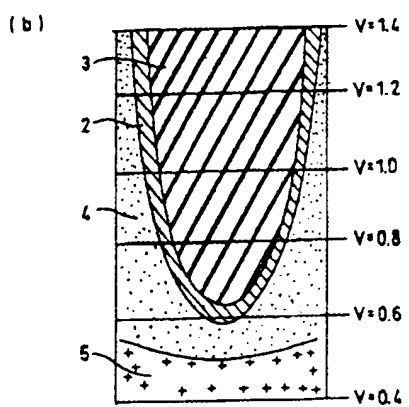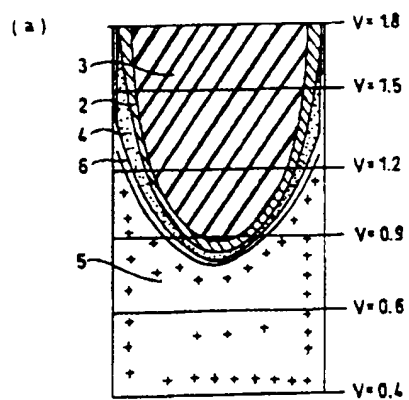horizontal axis: distance from center (mm)

[FIGURE 2]

[FIGURE 3]
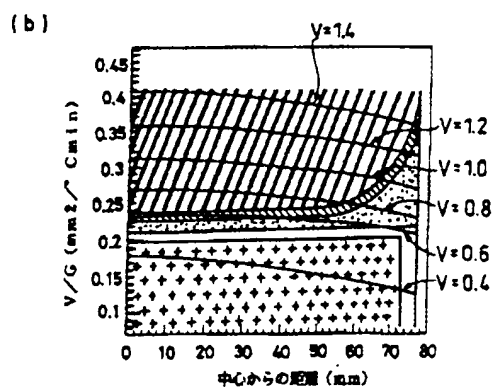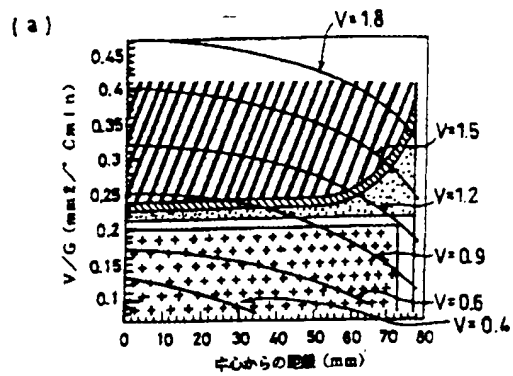

[translation of Japanese text in Figure 3]

horizontal axis: distance from center (mm)

[FIGURE 4]

(a)

3
2
4
6

5

V = 1.8
V = 1.5
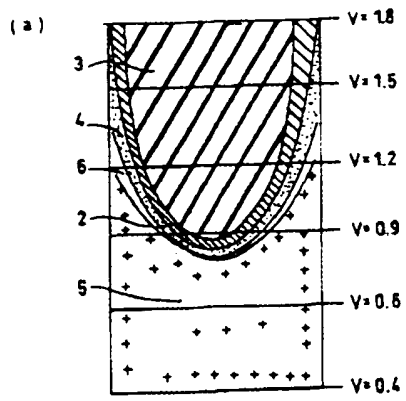V = 1.2
V = 0.9
V = 0.6
V = 0.4

(b)

3
2
4

5

V = 1.4
V = 1.2
V = 1.0
V = 0.8
V = 0.6
V = 0.4

[FIGURE 5]

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(a)

V=1.8
V=1.5
V=1.2
V=0.9
V=0.6
V=0.4

V/G (mm2/Cmin)

(b)

V=1.4
V=1.2
V=1.0
V=0.8
V=0.6
V=0.4

V/G (mm2/Cmin)

[translation of Japanese text in Figure 5]
horizontal axis (both): distance from center (mm)

[FIGURE 6]

(a)

```
3
4
6
2
5
```

V = 1.8
V = 1.5
V = 1.2
V = 0.9
V = 0.6
V = 0.4

(b)

```
3
4
2
5
```

V = 1.4
V = 1.2
V = 1.0
V = 0.8
V = 0.6
V = 0.4
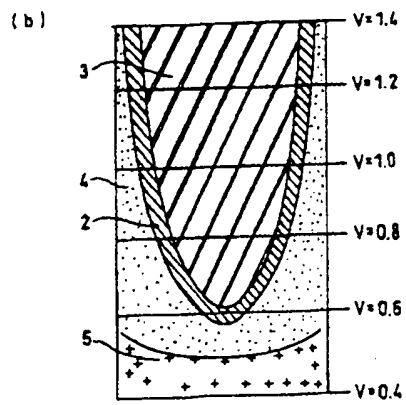
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